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A New Technique for the Simultaneous Measurement of Thermal Diffusivity and Thermal Conductivity of Small Energetic-Material Specimens

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Abstract

An experimental technique to simultaneously measure the thermal diffusivity and thermal conductivity of small specimens of thermal-insulator materials is described. Experimental conditions that approach the mathematical idealization of a one-dimensional, infinite, two-component, composite solid whose planar interface is subjected to a step-function heat flux are developed. The heat flux is generated by resistive heating of a thin metallic foil. Although this principle has been exploited previously, the present technique permits specimens with volumes two orders of magnitude smaller than prior work based on the same idealization. A number of difficulties related to small specimen volume are identified and overcome. Another improvement over past efforts is the use of nonlinear least-squares analysis to determine the optimum values for diffusivity and conductivity. A comparison of measurements of these properties for acrylic and Pyrex glass with literature values suggests that the technique affords accuracies of about 5% for materials in this range.

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1. INTRODUCTION

In connection with a program to measure and interpret temperature profiles in deflagrating propellants, there arose a need in our laboratory for the measurement of thermal conductivity using small specimens of solid propellant with dimensions of the order of half a centimeter. Steady-state measurements of thermal conductivity by the guarded-hot-plate method typically employ specimens as much as a hundred times larger[1] and are time-consuming because of the time required to establish steady-state conditions at each test temperature. As have many before us, we chose rather to pursue a transient-response experiment which, in addition to mitigating the drawbacks of the steady-state method, offers the advantage of simultaneously determining both the thermal diffusivity and thermal conductivity. This report describes the development of the technique and discusses the experimental subtleties arising from the use of such small specimens.

In measurements of thermal diffusivity, experimental conditions are usually arranged to approach the set of boundary conditions for which an exact solution is available to the following approximate form of the time-dependent, one-dimensional, heat-conduction equation:

$$\frac{\partial T}{\partial t} = \alpha \frac{\partial^2 T}{\partial x^2} \ , \tag{1}$$

where α is the thermal diffusivity, T the temperature, x the spatial coordinate, and t the time. In addition to having to satisfy the boundary conditions, the temperature range encompassed by the experiment must be small enough to neglect temperature variations of both the thermal conductivity (a condition required for validity of Equation (1)) and diffusivity (required for the validity of practically all the exact solutions).

In the flash method[2], for instance, the experiment is intended to simulate a homogeneous, insulated, finite-thickness slab of specimen material of infinite lateral extent, one face of which is instantaneously elevated in temperature. The temperature increase is accomplished by exposing the sample, whose face is blackened to boost the amount of energy absorbed, to the radiant output of a

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flash tube or pulsed laser. Developed for metals, the experimental times are short enough that conductive and convective heat losses at the sample boundaries can be ignored. Radiative heat losses are important at high temperatures, and correction strategies have been discussed by Clark and Taylor[3]. Conduction and convection losses at the sample boundary are important for low-diffusivity materials but can be virtually eliminated by placing the sample in a vacuum chamber[4]. The method has been found suitable for sample sizes down to 6 mm.

A simpler experimental setup was used by Plummer, et al.[5] for various glasses/ceramics and by Harmathy[6] for building materials such as bricks and concrete blocks. Both articles describe the use of resistively heated metal foils in an arrangement intended to approximate a semi-infinite, one-dimensional solid whose surface is subjected to a constant, spatially uniform heat flux of sudden onset. Both studies also used specimens whose lateral dimensions were on the order of 10 cm. The choices of experiment duration, temperature-sampling distance, foil thickness, heating power, etc. were conditioned by the availability of relatively large specimens and the desire to minimize errors in the diffusivity measurement. Steere[7] showed that Harmathy's technique could be extended to specimens consisting of multilayers of thin-film polymers and to specimens about two orders of magnitude smaller in volume, mainly by using very thin metal foils. In this paper we further extend the technique to specimens about two orders of magnitude smaller in volume than those used by Steere[7].

The present effort was undertaken in the belief that a heated-foil source would require less sample-preparation labor and utilize more readily available equipment than the radiant flash method. First efforts concentrated on impulse heating of the ribbon by capacitor discharge. It was found, however, that conductive heat losses from the specimen to the ribbon subsequent to the pulse were important and could not be quantified sufficiently to permit an accurate determination of the thermal diffusivity. Step-function heating of the foil eliminated this difficulty. A number of problems arose in connection with the small size of our specimens. In the discussion to follow, we argue that experimental conditions have been established for reasonably accurate simultaneous determinations of thermal diffusivity and thermal conductivity in very small specimens. In addition, a novel data

reduction strategy based on a nonlinear least-squares analysis is presented.

2. IDEALIZATION

Equation (1) has the following exact solution[8] for the semi-infinite region $x > 0$ upon imposition of a constant heat flux f_0 for $t > 0$ at $x = 0$:

$$T = T_0 + 2\frac{f_0}{\lambda}\sqrt{\alpha t}\ \mathrm{ierfc}\left(\frac{x}{2\sqrt{\alpha t}}\right)\ ,$$

(2)

where $T = T_0$ for $t < 0$ at all x, and $T = T_0$ for all t at $x = \infty$. The complementary error-function integral is defined as[8]

$$\mathrm{ierfc}(z) \equiv \sqrt{\frac{1}{\pi}}\,e^{-z^2} - z\,\mathrm{erfc}(z)\ ,$$

(3)

and the complementary error function is defined as

$$\mathrm{erfc}(z) \equiv \frac{2}{\sqrt{\pi}}\int_z^{\infty} e^{-\xi^2}d\xi\ .$$

(4)

The thermal diffusivity α is defined by

$$\alpha \equiv \frac{\lambda}{\rho c_p}\ ,$$

(5)

where λ is the thermal conductivity, ρ is the mass density, and c_p is the specific heat at constant pressure.

The experiment to simulate this theory must guarantee that the heat wave is one-dimensional at the temperature-sampling distance x, that the total specimen thickness is sufficiently large that the temperature at x is unchanged by further thickness increases, and that the heat flux entering the

specimen adequately approximates a mathematical step function. Additionally, instrumentation of the specimen to measure temperature transients at x must not jeopardize its monolithic quality and be adequately responsive. These issues are addressed in subsequent discussion.

3. EXPERIMENTAL ARRANGEMENT

Figure 1 is a cross-sectional schematic of the fixture developed to measure the thermal diffusivity and conductivity. Specimens are cut from solid cylindrical material of about 6.5-mm

Specimen Tensioning Screw

Guard Tensioning Spring

Specimen Guard

Specimen (Back-Up)

Foil Thermocouple

Terminal Block

Constantan Foil

Specimen (Wafer)

Foil Guard

Macor Insulator

Pedestal

Temperature-Controlled
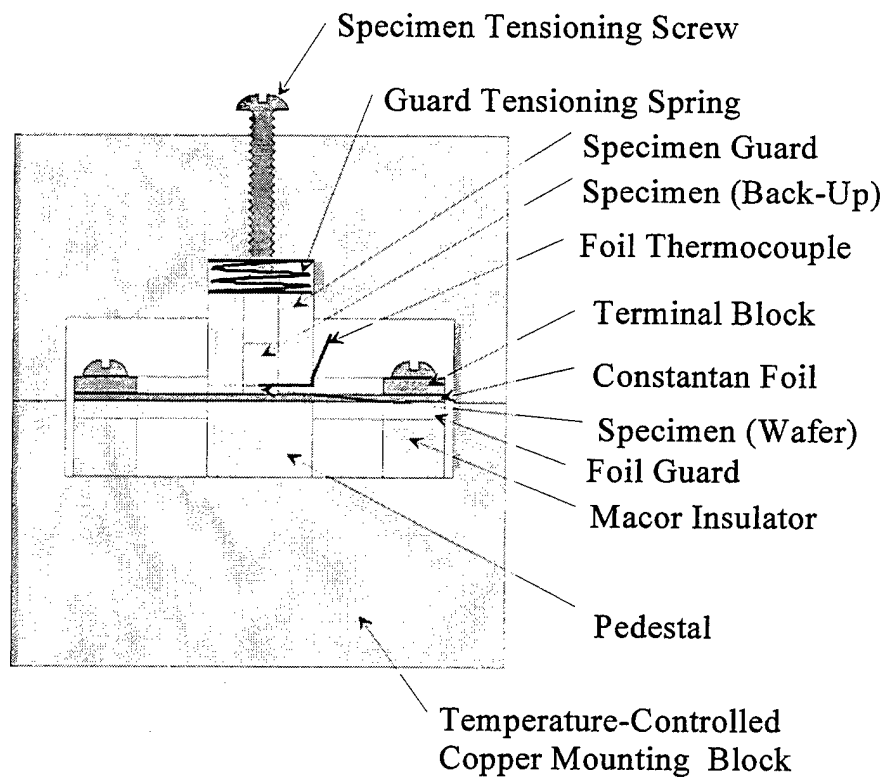Copper Mounting Block

Figure 1. Schematic of thermal diffusivity/conductivity fixture in cross-sectional view.

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diameter using a diamond-bladed, precision wafering saw. This saw operates at low speed using water as a cutting fluid, producing low-deformation cuts and variations in thickness for a single wafer of less than 1%. A complete specimen consists of a specimen wafer usually between 0.5 and 2.0 mm thick and a specimen back-up piece about 6.4 mm long. The thickness of the specimen wafer is measured with a micrometer which has a resolution of 0.0025 mm. Temperature is measured between the wafer and back-up piece by means of a bare Chromel/Alumel foil thermocouple of 0.005-mm thickness. Care is taken to place the thermocouple junction at the center of the wafer. The transient signal is amplified by a differential amplifier set to gain of 1,000 and captured by a digital oscilloscope. A typical trace consists of almost 2,000 data points.

On one of its sides the foil is in contact with the specimen wafer, and on the other it contacts an acrylic pedestal or base. In this respect our setup differs from that of Plummer, et al.,[5] Harmathy,[6] and Steere,[7] all of whom placed specimen material on both sides of the foil in a symmetric arrangement. The pedestal, so long as it is sufficiently broad and thick, does not affect the functional dependence of the temperature on α, x, and t but does affect the dependence of the temperature on the specimen conductivity, λ. The mathematical solution for the case of the infinite composite solid is affected by the thermal diffusivity, α_b, and thermal conductivity, λ_b, of the pedestal or base material as follows:[9]

$$T = T_0 + \left(\frac{2f_0 \lambda \sqrt{\alpha_b}}{\lambda \sqrt{\alpha_b} + \lambda_b \sqrt{\alpha}} \right) \sqrt{\alpha t} \; \text{ierfc} \left(\frac{x}{2\sqrt{\alpha t}} \right) . \tag{6}$$

Equation (6) is the governing equation for this work. The heat flux f_0 generated in a foil of resistance R, length L, and width W through which a current I is passing is given by

$$f_0 = \frac{I^2 R}{LW} . \tag{7}$$

The foil employed here is 5-μm-thick Constantan,[10] although 5-μm and 13-μm Manganin foils were also tested in the early development stages. Manganin has a somewhat lower resistivity temperature

coefficient but more readily tarnishes. Although we have no evidence that this thin oxide layer affects the experiment, it seemed prudent to avoid the issue by utilizing Constantan.

4. EXPERIMENTAL CONDITIONS FOR SATISFYING THE IDEALIZATION

In this section the extent to which the experiment conforms to the mathematical idealization embodied in Equation (6) is examined in detail.

4.1. Step-Function Flux

The mathematical ideality of the step-function heating stimulus is influenced by the finite rise time of the current in the foil and by the finite heat capacity of the foil. The rise time of the current was measured with a Pearson coil to be about 3 ms when switching was accomplished by a mercury-wetted relay, and no ringing was observed in the waveform at the edge of the step. With a total data-acquisition time of 4 s, such a rise time is seen to be very abrupt. A standard toggle switch was used initially, but the switching time was found to be about 30 ms and noisy, probably due to contact bounce. A regulated DC voltage supply provided power to the foil. The voltage drop across the foil proved constant to within about 0.1%. Thus, the step-function character of the *current* flowing in the foil would seem to be quite well approximated experimentally. However, even if the current were a perfect step function, the finite heat capacity of the foil results in a delay in achieving a constant heat flux into the specimen. This delay is longer for thicker foils, and its effect on the determination of diffusivity is greater the shorter the data acquisition time and the lower the diffusivity of the test specimen. An exact solution to this problem has been derived using Laplace transforms, and its impact on thermal diffusivity measurement discussed in detail elsewhere.[11] For our experimental conditions, this source of error results in less than a 2.5% underestimate of the diffusivity. As this error is smaller than the estimated accuracy of the technique developed here, no correction seems warranted although it is certainly feasible. One could also further minimize the effect by using even thinner foil.

An interesting problem arose in computing the heat flux by Equation (7). At first the flux was computed by measuring the voltage drop across the terminal blocks holding down the foil at each end. This voltage drop was then multiplied by the current flowing in the circuit to compute the power dissipated in the foil. Unfortunately, when the ratio of the diffusivity to the conductivity, both measured in the experiment, was compared to the product of independently measured density and specific heat as a consistency check (see Equation (5)), it was found that the former ratio was consistently about 20% smaller than the latter ratio. The accuracy of the ρc_p product was about 5%, and thus could not account for the discrepancy. This problem turned out to be due to electrical contact resistance between the terminal block and the foil. Measurements of the total resistance between terminal blocks (voltage drop across the terminal blocks divided by current) as a function of foil length are shown in Figure 2.

The intercept is the contact resistance, determined by the least-squares fit to be about 0.06 Ω. Since the foil resistance for our standard 4.4 cm long foil is only 0.336 Ω, about 15% of the power to the terminal blocks is dissipated by the contact resistance between terminal block and foil. The magnitude of this effect was not anticipated, and it proved difficult to diagnose. Once recognized, however, the problem was remedied by using in Equation (7) the actual foil resistance, which can be determined from the slope of the line in Figure 2. This problem was not encountered by
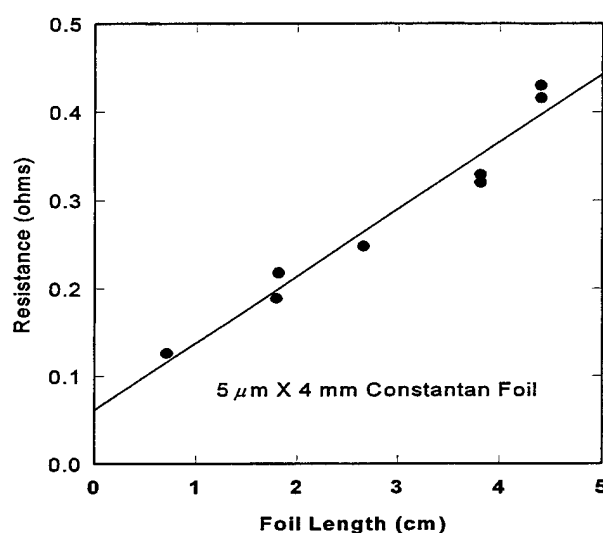

Figure 2. Measurements of total electrical resistance of series circuit containing Constantan foil and contact resistances between foil and terminal blocks for foils of varying lengths. Total electrical contact resistance is the y-intercept of the least-squares fit (solid line).

Harmathy[6] or Steere[7] because of their much larger specimen (and therefore foil) sizes.

4.2. Uniformity of Flux

In addition to the temporal constraints on the heat flux, the mathematical idealization also presumes that the heat flux is spatially uniform (in the plane of the specimen surface) in order to assure one-dimensionality. A number of experimental details have a bearing on this issue. The foil used in our fixture is about 1.2 cm wide by 4.4 cm long, with the long dimension being the distance between the electrical terminal blocks. The terminal blocks are made of relatively massive (0.3 cm thick) flat copper bar stock and therefore act as a heat sink at the ends of the foil for heat generated along the length of the foil. If the length of the foil is too short, the flux into the specimen will not be spatially uniform due to temperature gradients at the ends of the foil in contact with the terminal blocks. One may address this problem by examining an exact solution for the limiting case of no heat loss from the surface of the foil and terminal-block temperatures maintained at ambient temperature. This case would be a poor assumption to make if one were trying to model the actual temperature distribution in the foil; however, such a case overestimates the heat conduction to the terminal blocks, thereby giving useful worst-case design guidance. The solution[12] for the temperature difference from ambient at some distance y from one end of a conductor of length L and thermal diffusivity α_c consists of the sum of a steady-state term and a transient term. Dividing this solution by the adiabatic solution gives the fraction, F, of the foil temperature at the given distance y that one would expect in the absence of heat loss to the terminal blocks, i.e.,

$$F = \frac{y(L-y)}{2\alpha_c t} - \frac{4L^2}{\pi^3 \alpha_c t} \sum_{n=0}^{\infty} \frac{\exp\left[-\frac{\alpha_c(2n+1)^2\pi^2 t}{L^2}\right] \sin\left[\frac{(2n+1)\pi y}{L}\right]}{(2n+1)^3} \quad . \tag{8}$$

This fraction is plotted for a Constantan foil at several times in Figure 3. One can see that the assumption of temperature uniformity along the length of the foil becomes worse with time but is

quite good, even under our worst-case conditions, before 5 s in the center of the foil over the width of our specimen. Thus one may be quite assured of the flux uniformity along the length of the foil due to heat loss to the terminal blocks during our adopted data-acquistion period of 4 s.
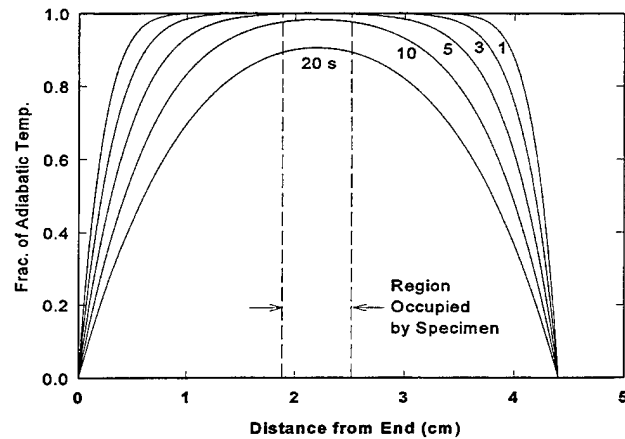

Figure 3. Worst-case calculations of nonuniformity in foil temperature due to heat losses to terminal blocks at the ends of the foil. Temperatures are expressed as a fraction of the temperature of the foil if there were no heat losses. Nonuniformities in foil temperature over the region occupied by the specimen will result in spatial nonuniformity of the heat flux, invalidating the mathematical idealization.

Edge effects across the width of the foil are another source of nonuniformity in the heat flux. Along a line perpendicular to the plane of the foil and through its center, the flux appears one-dimensional for distances sufficiently close to the foil. At distances from the foil that are large compared with its width, the flux appears to emanate from a line source (assuming the foil is sufficiently long). We again examine this aspect by means of an exact solution for worst-case boundary conditions. Consider the temperature along a line perpendicular to and through the center of a circular disk from which a step-function heat flux emanates. Two-dimensionality in the resulting temperature field will clearly be evidenced at closer distances than in the case of the rectangular foil, making this a valid worst case. The solution for the temperature along the perpendicular through the center of the disk source[13] ratioed with the one-dimensional solution (Equation (2)) is plotted in Figure 4. Results are shown for both acrylic and fused-quartz substrates. Deterioration of one-dimensionality occurs at much earlier times for acrylic, but at the end of our data-acquisition time of 3.6 s, the temperature at $x = 0.13$ cm is still 99% of the idealized one-dimensional value. Bearing in mind that this is a worst-case comparison, it is clear that foil edge effects can have but a negligible influence on results for our experimental conditions.

A third reason for spatial nonuniformity of the heat flux relates to the small size of the specimen itself. Our specimens are in the form of right circular cylinders with a diameter of about 6 mm. If the specimen is placed at the center of the foil, there is uncovered foil surrounding the specimen. This unbalanced boundary condition leads to temperature gradients in the plane of the foil radial to the axis of the specimen. This problem is remedied by the use of a guard material surrounding the specimen and covering the foil up to the terminal blocks. In principal one should use a guard material with thermal properties identical to the specimen itself. In practice we have found that use of an acrylic guard results in acceptable accuracy for Pyrex glass, for example, which has a thermal diffusivity of about six times that of acrylic. Tests using an acrylic specimen wafer showed that the temperature difference from ambient after 5 s is only about 2% lower at the periphery of the specimen than at its center when a 5-µm-thick foil is covered by the guard. This result compares to temperature drops of 18% from center to edge of the specimen in the absence of a guard.

Figure 4. Worst-case calculations of departures from one-dimensionality of the temperature field in the specimen due to the finite width of the foil for two different specimen materials. Calculated is the temperature generated at a point along an axis through a disk source of diameter equal to the width of the foil. The results are displayed as a fraction of the temperature generated by a planar source of infinite extent.

4.3. Semi-Infinite Specimen

The condition that the sample be sufficiently thick as to approximate being infinite may be determined as follows. The solution of Equation (1) for a slab of finite thickness l (and infinite lateral dimensions) subjected to step-function heating is[14]
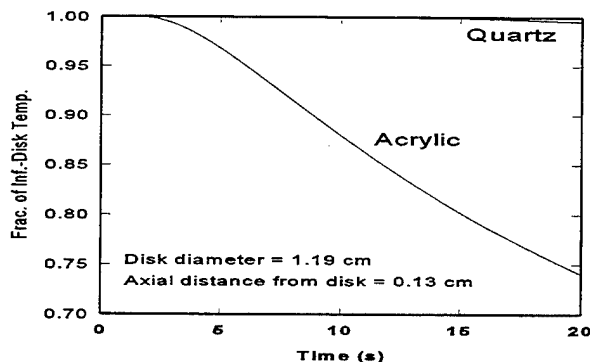
$$T = T_0 + 2\frac{f_0}{\lambda}\sqrt{\alpha t}\sum_{n=0}^{\infty}\left\{\text{ierfc}\left[\frac{2nl+x}{2\sqrt{\alpha t}}\right] + \text{ierfc}\left[\frac{2(n+1)l-x}{2\sqrt{\alpha t}}\right]\right\} \quad . \tag{9}$$

For a given temperature-sampling station x_1 and a given experiment duration t_1, there will be some minimum slab thickness l_1 beyond which the temperature at x_1 by Equation (2) will differ from that by Equation (9) by no more than, say, 1 %. For times earlier than t_1 the temperature differences between the two equations are even less than 1 %. Figure 5 illustrates the point quantitatively for $x_1 = 0.3$ mm and a 1 % criterion. If the diffusivity of the sample is 1.0×10^{-3} cm^2/s and the data-acquisition time is 4 s, then by Figure 5 the minimum sample thickness l_1 required to satisfy the infinite-thickness boundary condition is a little over 1 mm. If either the time or the diffusivity is larger, then a thicker

Figure 5. Calculations of the minimum thickness of a specimen (wafer + back-up piece) for which the temperature at a distance from the foil equal to the indicated wafer thickness differs from the temperature that would result from an infinite-thickness specimen by 1%. The figure shows, for example, that a total specimen thickness of 0.6 cm is sufficiently thick to approximate being infinitely thick in testing a material with a thermal diffusivity of 0.01 cm^2/s if the data acquisition time is 10 s (or less). Vertical decade grid lines are shown for convenience.

sample would be required. In this work the temperature-sampling distance x_1 is always less than 0.3 mm, the sample thickness is 6 mm or greater, and the data-acquisition time is 4 s. Thus, from the standpoint of sufficient sample thickness alone, the experimental setup is suitable for thermal diffusivities up to 0.2 cm^2/s.
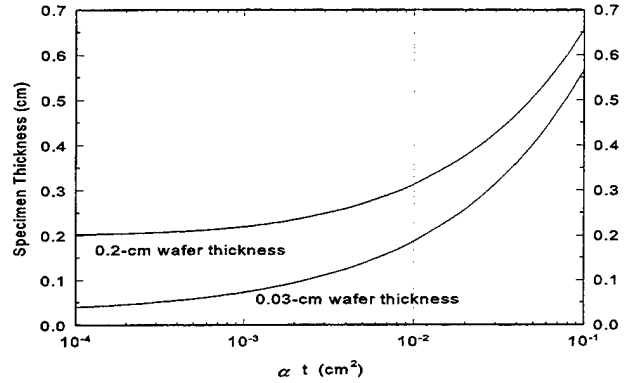
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D. Instrumentation Issues

At the outset of this study it was thought that the effects of thermal contact resistance might be significant at the specimen interface which includes the thermocouple and at the foil-specimen interface. In order to test the effects of contact resistance on the deduced value of diffusivity, three experiments were performed. In the first, an extra interface was introduced between the foil and the temperature-sampling station by stacking two wafers. The diffusivity was measured for this setup and compared to the value obtained using a single wafer of a thickness approximately the same as the combined thickness of the two wafers. In a series of five tests each, the difference in average values was not statistically significant (i.e., they differed by an amount less than or equal to the standard deviation of the mean). In the second experiment, an additional interface was introduced beyond the temperature-sampling station (i.e., at larger x). This modification, likewise, did not affect (in the same statistical sense as previously mentioned) the diffusivity values obtained in a five-test series. In the third experiment, conductive paste was used at each interface, but the diffusivity and conductivity results were unchanged (again relative to the standard deviation of the mean). These experiments prove that errors arising from thermal contact resistance in these low-diffusivity materials are insignificant. This same conclusion was reached by Shelley and Huber[15] and by Steere[7].

Another potential source of error is the adequacy of thermocouple response. A convenient way to address this problem is to compare results using two different thermocouple sizes. We found that Chromel/Alumel foil thermocouples, 5 μm and 12 μm thick, produced average diffusivity and conductivity values differing by statistically insignificant amounts. These tests were performed using the acrylic specimen in preference to fused quartz since the temperature gradients are higher in acrylic, resulting in greater sensitivity of the measurements to thermocouple response.

No measurable differences in diffusivity/conductivity could be attributed to variations in the specimen stack tension controlled by the tensioning screw at the top of the fixture. This tension was

normally set to bring the components into intimate contact without deforming or breaking the specimen components.

The magnitude of the heat flux is controlled by the voltage across the foil supplied by a regulated DC power supply. The choice of heat-flux magnitude was a compromise between a desire, on the one hand, to maximize the signal-to-noise ratio (improved by high fluxes) and the requirement of Equation (6), on the other hand, that the diffusivity and conductivity of the sample remain constant during measurement. A flux f_0 of about 0.25 watts/cm^2 was found to be an acceptable compromise between these limits, although a wide range of powers was found to be permissible.

The period of data acquisition is likewise a compromise between competing requirements. Short times are desirable to minimize radial heat losses and effects related to the finite length of the specimen, but long times are desirable from the standpoint of signal-to-noise ratio. The compromise adopted was 4 s, partitioned into an initial-temperature data period for the first 0.4 s and a heating phase lasting 3.6 s. This period is also consistent with the avoidance of nonuniform heat flux arising from the finite length and width of the foil as just discussed.

5. DATA ANALYSIS

A number of approaches might be taken to obtain a value for the thermal diffusivity from the measured temperature history between the specimen wafer and back-up piece. Plummer, et al.[5] measured the ratio of the temperature at some x to that of the foil, then obtained the diffusivity graphically. Harmathy[6] ratioed the temperature at a single position and at two different times, then found α graphically. The method of analysis used in the present work is based on the temperature measured at a single x but utilizes temperature data at all times during the experiment. This more comprehensive approach is enabled by performing a nonlinear least-squares fit[16,17] of Equation (6) to the data using α and λ as fitting parameters. Because of noise in the data, it proved desirable to include T_0 in the list of fitting parameters rather than to input an averaged value. Likewise, to

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minimize the effects of measurement imprecision in wafer thickness on the deduced value of diffusivity, five measurements were made of wafer thickness over its surface, and these values were included in the fitting process. In all cases the fitted values of T_0 and x were very close to their separately averaged values. In setting up the experiment, the digital oscilloscope's pretrigger function is selected at about -0.4 s. This results in the first N_{T_0} points being captured while the specimen is still at the initial temperature T_0 and the next N_T points captured during the constant heating phase at $t>0$. If there are N_x wafer thickness measurements, then the fitting parameters, $\hat{x}$, $\hat{\alpha}$, $\hat{\lambda}$, and $\hat{T}_0$, are varied until the variance of the fit σ_{fit}^2, as given in the following equation, is minimized:

$$\sigma_{fit}^2 = \sum_{i=1}^{N_T} w_i^T \left(T_i - T(\hat{x},\hat{\alpha},\hat{\lambda};t_i) \right)^2 + \sum_{i=1}^{N_{T_0}} w_i^T \left(T_{0_i} - \hat{T}_0 \right)^2 + \sum_{i=1}^{N_x} w_i^x \left(x_i - \hat{x} \right)^2 \ . \qquad (10)$$

The weights are defined as the reciprocal of the respective parameter measurement variances, which are determined from experience. Note that this approach is not equivalent to inverting Equation (6) for α and λ using the average values of x and T_0. Not only are optimum values of α and λ obtained, consistent with the assumption of random error in the measurement of T, T_0, and x, but a more accurate estimate of the standard deviation in the fitted parameters is also available, which properly reflects the propagation of
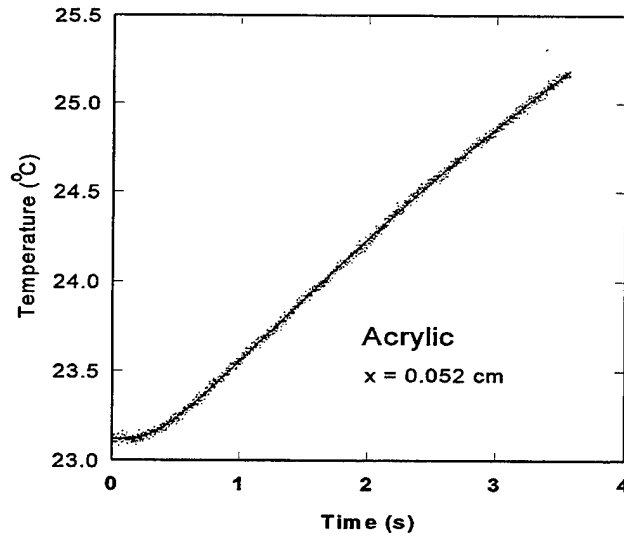

Figure 6. Typical experimental temperature data (points) measured for an acrylic (PMMA) specimen at a distance of 0.052 cm from the foil compared with the theoretical temperature (solid curve) computed from Equation (6) with the optimized values for thermal conductivity and diffusivity.

measurement errors through the nonlinear governing equation. Figure 6 illustrates a typical

comparison between Equation **(6)**, evaluated using the least-squares values of α, λ, x, and T_0, and the experimental data. The quality of the fit is seen to be excellent.

6. DIFFUSIVITY RESULTS

The technique just described was applied to samples of acrylic, Pyrex glass, and fused quartz at room temperature. The acrylic, poly(methylmethacrylate) or PMMA, was the same material from which the guard components were fabricated. The results for these materials compared with literature values are given in Table 1.

Fused quartz comes as close as any material to being a standard reference material in the lower conductivity range. In fact, the TPRC compendium[18] compiles enough data on the thermal conductivity of fused quartz to provide a set of recommended values, to an accuracy estimated as ±3%, through the temperature range of interest in this study. The subsequent work of Sugawara[19] claims to have measured the thermal conductivity of very pure fused quartz to an accuracy of ±1.5%. The values of diffusivity in Table 1 attributed to these two sources are obtained using our measured values of the density and specific heat of the fused-quartz specimen, viz., $\rho = 2.194$ g/cm^3 and $c_p = 0.175$ cal/g-K at 296 K. The present technique yields a value for the conductivity of fused quartz about 13% lower than those of the TPRC[18] and Sugawara[19]. The sample of fused quartz tested here is the lowest grade material manufactured by Heraeus Amercil (T08 Commercial Rod). It is not known to what extent its chemical impurity and bubble inclusions are responsible for the shortfall in conductivity just noted; however, Sugawara[19] identified sample purity as an important factor in the ±8% variation in conductivity values among different researchers. The tolerance values attached to our values are indicative of the precision of our measurement and are one standard deviation of the mean, typically of five separate runs.

Our measurements of the thermal conductivity for Pyrex glass (Corning #7740) differ by only 3% from the TPRC recommended values, lending confidence to our values and reinforcing suspicion

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that impurities in our fused-quartz specimen may be responsible for the relatively unfavorable comparison to the TPRC[18] values for that material.

TABLE 1. Results of Present Technique Compared to

Published Work on Similar Materials

TEST MATERIAL	10^3 x α (cm^2/s)	10^4 x λ (cal/cm-s-K)	REFERENCE
acrylic (PMMA)	1.14 ± .02 @ 296 K	4.29 ± .05 @ 296 K	this work
"	1.28 @ 296 K[‡]	-	Luikov, et al.[20]
"	1.13 @ 298 K	4.58 @ 298 K	Krischer & Esdorn[21]
"	0.948[†] @ 296 K[‡]	3.64 @ 296 K[‡]	Lucks, et al.[26]
"	1.24[†] @ 313 K	4.61 @ 313 K	Brown & Otten[22]
"	1.22[†] @ 293 K	4.52 @ 293 K	Eiermann[23]
Pyrex	6.45 ± .22 @ 296 K	25.4 ± 1.3 @ 296 K	this work
"	-	26.2 @ 296 K[‡]	TPRC[18] recommend.
"	5.5 @ 298 K	-	Plummer, et al.[5]
"	5.75 @ 298 K	23.9 @ 298 K	Hasselman, et al.[24]
fused quartz	7.72 ± .10 @ 296 K	28.6 ± 1.1 @ 296 K	this work
"	8.54[†] @ 295 K[‡]	32.8 @ 296 K[‡]	TPRC[18] recommend.
"	8.65[†] @ 295 K[‡]	33.2 @ 296 K[‡]	Sugawara[19]
"	8.33 @ 296 K[‡]	-	Luikov, et al.[20]
"	-	28.2 @ 314 K	Ballard, et al.[25]

[†] Computed from our measured value of ρc_p using Equation **(5)**.

[‡] Value at this temperature obtained by linear interpolation.

The literature values of α and λ for acrylic are more widely scattered and difficult to assess. No recommended values are offered in the TPRC[18] compendium. Although the references quoted

for acrylic are not comprehensive, we note that our value is almost exactly the average of the quoted literature values. If the possibly anomalous value of Lucks, et al.[26] is omitted, our measured conductivity is 6% lower than the resulting average literature value. In computing the diffusivity for acrylic from published values of the thermal conductivity, we have used our measurements of the density and specific heat, $\rho = 1.181$ g/cm^3 and $c_p = 0.315$ cal/g-K at 296 K.

Using our independent measurements of the mass density (estimated accuracy = 1%) and specific heat (estimated accuracy = 5%) for our acrylic and fused-quartz specimens, determined by dimensional measurement/weighing and differential scanning calorimetry, respectively, the self-consistency between diffusivity and conductivity yielded by the present experiment may be tested. For example, the measured value of conductivity differs from that computed from Equation (5) by 3% or less for acrylic and fused quartz. Taking all of the aforementioned comparisons into account, we therefore feel justified in claiming an accuracy of about 5% in measurements of the thermal diffusivity and conductivity by the present technique for materials falling into the range between acrylic and Pyrex glass. This same accuracy may also apply to fused quartz; however, it would be prudent to prove this by testing a specimen of purity comparable to that of Sugawara[19]. The solid-propellant materials which motivated this work fall quite generally in the more restricted range and therefore may suitably be studied using this technique.

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13. ABSTRACT (Maximum 200 words)

An experimental technique to simultaneously measure the thermal diffusivity and thermal conductivity of small specimens of thermal-insulator materials is described. Experimental conditions that approach the mathematical idealization of a one-dimensional, infinite, two-component, composite solid whose planar interface is subjected to a step-function heat flux are developed. The heat flux is generated by resistive heating of a thin metallic foil. Although this principle has been exploited previously, the present technique permits specimens with volumes two orders of magnitude smaller than prior work based on the same idealization. A number of difficulties related to small specimen volume are identified and overcome. Another improvement over past efforts is the use of nonlinear least-squares analysis to determine the optimum values for diffusivity and conductivity. A comparison of measurements of these properties for acrylic and Pyrex glass with literature values suggests that the technique affords accuracies of about 5% for materials in this range.

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